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SORPTION OF HYDROGEN BY GLASSES AND CERAMICS

Report No. 4

Fourth Quarterly Progress Report
1 November through 31 January 1964

Contract No. DA36-039-AMC-02170(E)
DA Task No. 1 G6-22001-A-055-02

13 February 1964

U. S. Army Electronics Research and Development Laboratories

Fort Monmouth, New Jersey

RUTGERS, THE STATE UNIVERSITY
School of Chemistry
New Brunswick, N. J.

SORPTION OF HYDROGEN BY GLASSES AND CERAMICS

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Fourth Quarterly Progress Report

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Objective:

To broaden the understanding of the cleanup of gases in gas-filled electronic devices, with equal emphasis being placed on the measurement of the kinetics of the process and the determination, using infrared spectroscopy, of the structure of molecular species formed by the gas-solid interaction, so that a mechanism that is both scientifically and technologically useful may be developed.

Contract No. DA36-039-AMC-02170 (E)

Technical Guidelines, Dated 8 August 1962

DA Task No. 1 G6-22001-A-055-02

Report prepared and approved by M J D Low

M. J. D. Low
Principal Investigator,
Assistant Professor of
Physical Chemistry

RUTGERS, THE STATE UNIVERSITY
School of Chemistry
New Brunswick, N. J.

1964

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PURPOSE

The purpose of this contract is to broaden the understanding of the cleanup of gases in gas-filled devices such as thyratrons and rectifiers. Cleanup appears to occur on all of the surfaces of the tube components, the extent of cleanup by any one component depending on the nature of that component. The mechanism of this phenomenon will be investigated.

The kinetics of the cleanup of hydrogen and of deuterium by glasses and ceramics will be measured, but equal emphasis will be placed on gaining an understanding of the mechanism of the cleanup by determining the molecular structures formed by the interaction of gases with the solids. This entails the use of infrared spectroscopic techniques that have previously been very successfully applied to the study of the structure of molecular species formed on the surfaces of catalytically-active solids.

Advances in our knowledge of both the kinetics of cleanup and of the species formed during the process is to be integrated with existing knowledge so that a mechanism may be deduced that will further scientific understanding and that can also aid in the alleviation of the cleanup problem.

ABSTRACT

Infrared spectra were obtained of the thermal decomposition of $Be(OH)_2$, of H_2-BeO interaction, and of the influence of Pt on the exchange of OH groups on alumina surfaces with D_2.

PUBLICATIONS, LECTURES, REPORTS, AND CONFERENCES

Publications: None

Lectures: None

Reports: The third quarterly report was submitted to USAELRDL, as required by the contract.

Conferences:

At USAERDL, Evans Area

3 December, 1963

Present: M. H. Zinn, S. Schneider, and N. L. Yeamans, UCAELRDL

M. J. D. Low, Rutgers, The State University

- - - - -

At Wright Laboratory, Rutgers, The State University, New Brunswick, New Jersey

29 January 1964

Present: J. Creedon, N. L. Yeamans, USAELRDL

M. J. D. Low, M. Courtois, N. Ramasubramanian, Rutgers, The State University, New Brunswick, New Jersey

FACTUAL DATA

1. Status of Work

The main experimental topics during this report period were concerned with the isotopic exchange of hydrogen with deuterium on the surface of pure alumina and of alumina containing some platinum, with the sorption of hydrogen by BeO, and with the decomposition of $Be(OH)_2$ to form BeO and the subsequent interaction of hydrogen with the decomposition product. Very little work was done on the topic of measuring gas uptake under activated conditions, because difficulties were encountered with measurements of the hydrogen atom concentration in the presence of an electrodeless discharge, and because of a temporary breakdown of the rf transmitter used for producing the discharge. These difficulties have caused some delay in the over-all plan of work, and have brought about the distribution of work effort on the various topics described.

2. Infrared Sample Cells

Some of the work on infrared spectra of alumina, BeO, and $Be(OH)_2$ samples described in later sections required high-temperature heat treatments of the samples <u>in vacuo</u>. This has caused us to design and build sample cells for infrared work in which a sample could be heated <u>in vacuo</u> or under controlled gas atmospheres to about 1000°C.

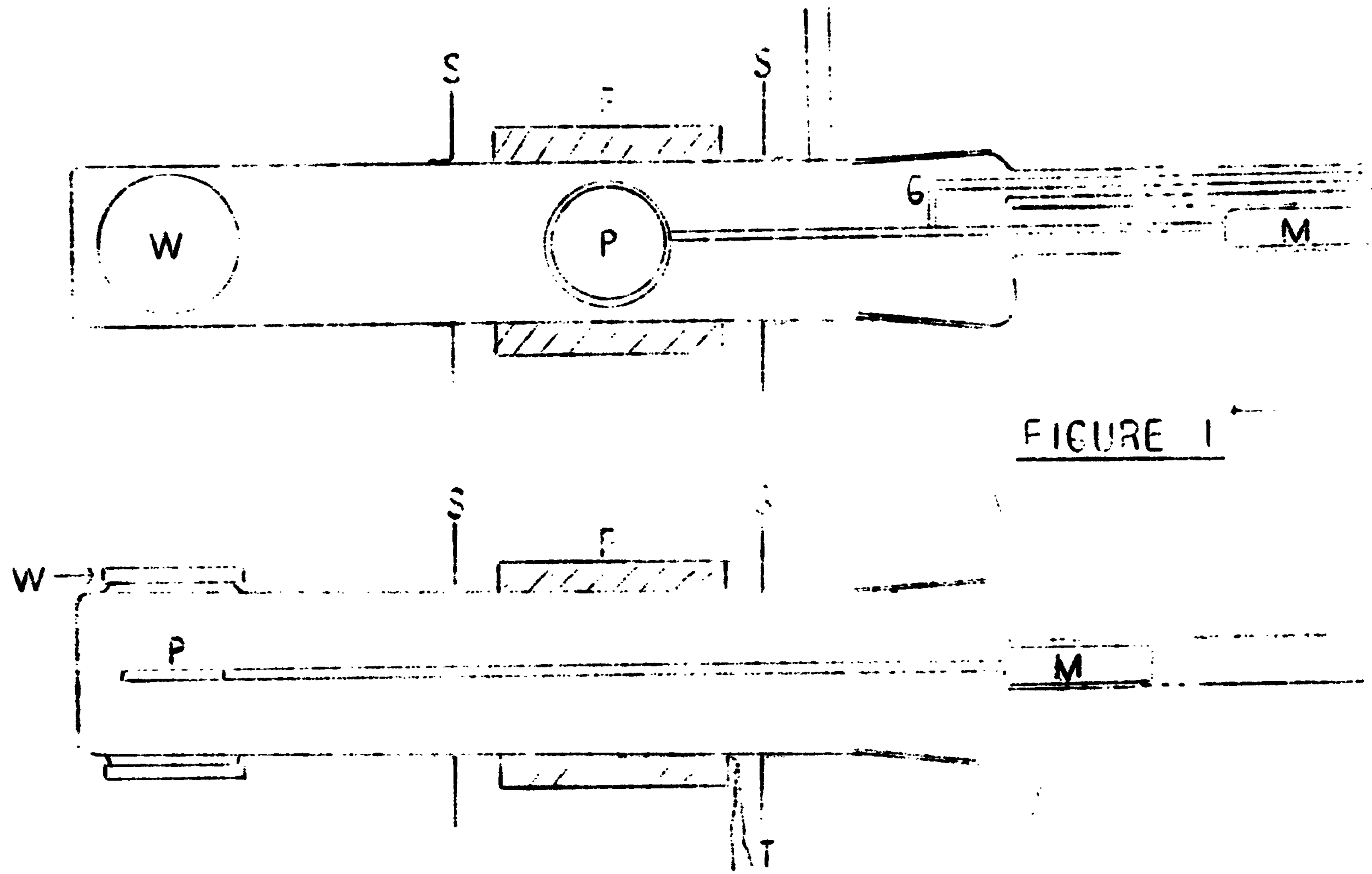


FIGURE I

HIGH TEMPERATURE IR CELL

As far as is known, there are no materials suitable for use as "windows" in infrared cells that can withstand high temperatures for any length of time. Also, the bonding materials used to make a vacuum-tight seal between existing window materials and cell bodies, or most cell bodies themselves, similarly cannot withstand high temperatures. Cells were therefore constructed in which the sample was moved from the region of the cell windows into a region of the cell body that could be heated. The cell bodies were made of Vycor.

Figure 1, facing, illustrates the simple design of the cells. A female $\$$ 34/40 Vycor joint was fused to a length of 32 mm O.D. Vycor tube closed at one end. The tubing was worked into the shape indicated by the side and top view of the cell body shown in Figure 1. Cell windows W were attached to the body with Glyptal. A furnace F consisting of nichrome wire and asbestos was wound on the body. Heat shields S made of household aluminum foil serve to protect the windows and the ground joint from excessive temperature rise. The sample pellet P rests in a quartz ring that is a part of a movable carriage made of quartz rod. The carriage is moved by the action an externally-applied magnet on a Vycor-enclosed magnet M attached to the carriage. The quartz guide rod G serves to keep the sample vertical. Temperature is monitored by means of thermocouple T. On operation of the cell up to about 1000°C (the temperature at which the furnace insulation and heating wires fail),

the windows and joint reach such a temperature that they become uncomfortable to touch. In order to obtain a spectrum, the carriage is moved so that sample P is situated between the windows W. The carriage is retracted so that P is within the furnace zone in order to perform a heat treatment of the sample.

3. Sorption Under Activated Conditions.

Attempts were made to start making measurements of the uptake of hydrogen on alumina in the presence of an electrodeless discharge, without success. In order to make meaningful measurements of gas uptake, some estimation of the intensity of the discharge within the gas must be made. This is being attempted by measuring the temperature rise brought about by the recombination of hydrogen atoms on a platinum filament within the gas. An electronic instrument was built during the third report period to measure the change in temperature, and hence of resistance, of the filament.

It was found that there was so much rf interference with the transistor circuits of the instrument that meaningful measurements could not be made. After considerable experimentation, involving improvement of the shielding of cables and the incorporation of condensors in the electronic circuits, the interference from the rf generator was eliminated. Preliminary trial of the instrument suggest that the desired measurements can now be made.

4. Sorption of Hydrogen by BeO

(a) Sorption Experiments

The sample BeO (II), which had already been used for the hydrogen adsorption studies at 300°C described in a previous report, was transferred to a Vycor tube attached to the adsorption system. The sample was degassed at 700° for 15 hours at 10^{-6}mm Hg. The degassed sample was then exposed to hydrogen at 300° at 1 cm Hg pressure. Little hydrogen uptake occurred. The experiments again showed that decreasing amounts of hydrogen were taken up on successive exposures to hydrogen, suggesting that a progressive change was occurring in the absorbent. The experiments were consequently stopped until further information would become available from improved spectroscopic studies.

(b) Infrared Spectra

Attempts to obtain infrared spectra of BeO (II) samples and of hydrogen chemisorbed on BeO (II) were continued. Three pellets of BeO (II) were examined after degassing at 400°, after being exposed to hydrogen at 300°, and after being exposed to hydrogen under discharge conditions. Evidence for bands caused by chemisorbed hydrogen was not obtained. In view of this, and because of the very low transmittance of the pellets, the experiments were stopped.

5. Interaction of Hydrogen with Decomposed $Be(OH)_2$

The eratic behavior of BeO samples obtained by decomposing the nitrate suggested the examination of BeO samples prepared by the thermal decomposition of $Be(OH)_2$. Pellets of $Be(OH)_2$ were prepared for infrared study, and the thermal decomposition of $Be(OH)_2$ *in situ* and the interaction of hydrogen with the product of the decomposition were studied at temperatures ranging from room

temperature to 850°C.

(a) Samples.

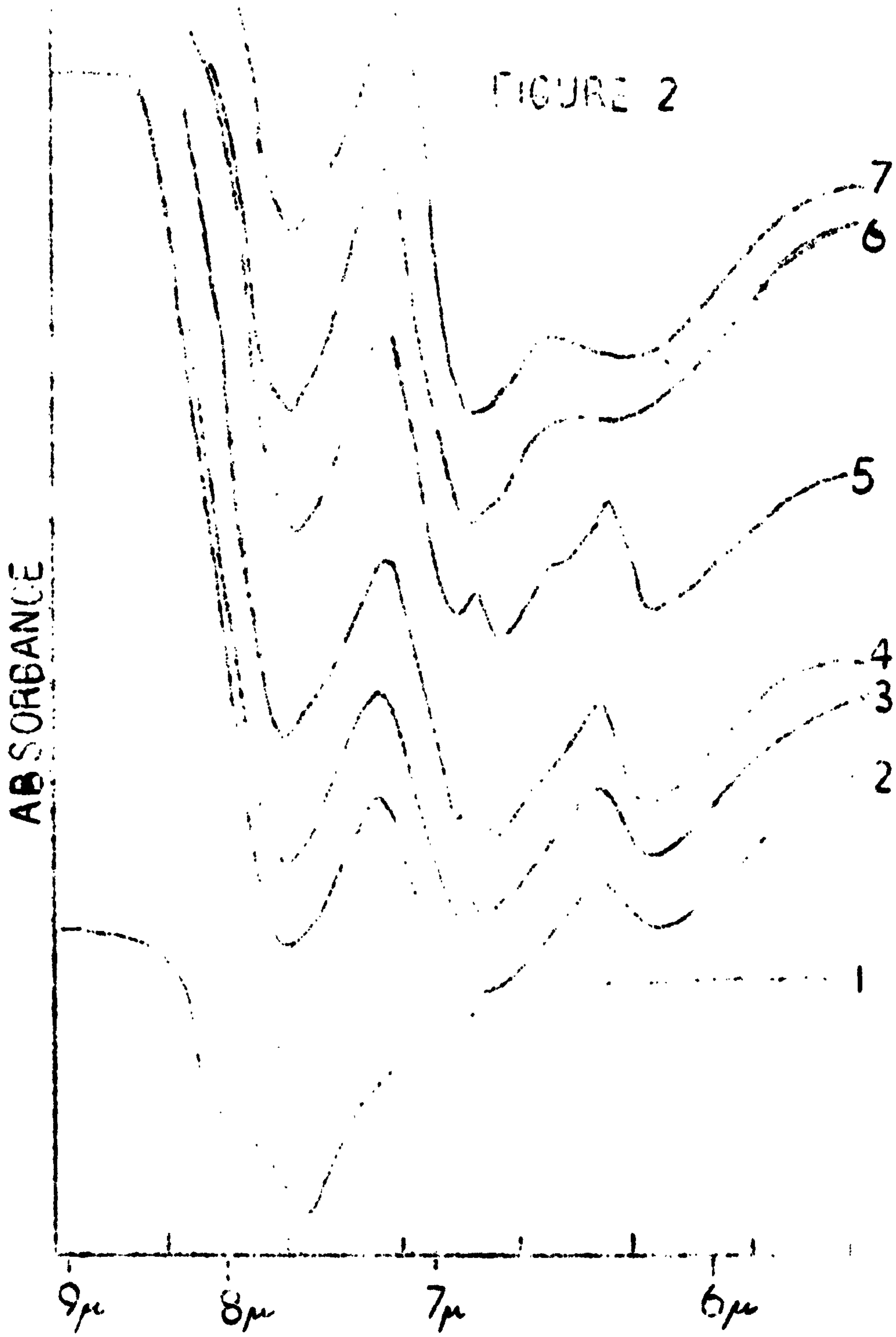
A total of 11 samples were prepared for examination. The sample pills must be thin, in order to permit a significant amount of infrared radiation to pass, and also must be mechanically strong, so that they may be attached to the sample holder and manipulated inside the infrared cell. All but three of the pellets placed in the cell did not survive more than initial spectroscopic investigation. Breakage occurred on transferring the sample to the furnace zone of the cell, or during heating or cooling. The fragmentary data obtained by examination of pellets which were broken are not considered here.

Pellets of 1 inch diameter were pressed in a hardened steel die at a pressure of 25 tons per square inch. Hard and brittle pellets of a porcelain-like appearance were obtained. All pellets were made of $Be(OH)_2$. The designation and weights of useful pellets were as follows:

$Be(OH)_2$-I... 0.07 gm
$Be(OH)_2$-VI.. 0.06 gm
$Be(OH)_2$-XI.. 0.10 gm

It had been intended to follow the thermal decomposition of $Be(OH)_2$ from room temperature to 500°C in a Pyrex cell. The spectra shown in (b) below indicated that higher temperatures were necessary. Consequently, a Vycor cell was built. The change of cells and breakage of pellets has caused three batches of overlapping data.

FIGURE 2



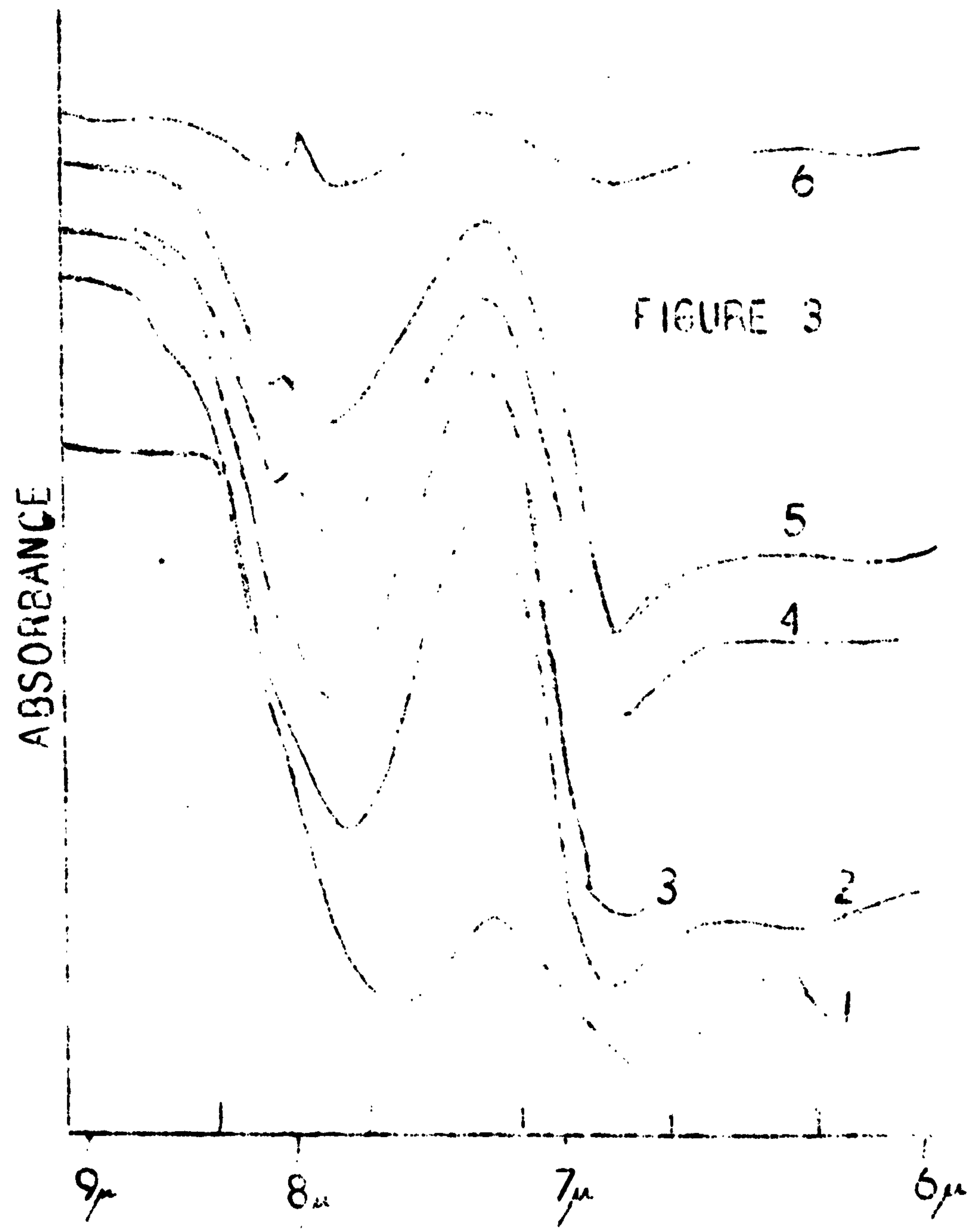
IR SPECTRA OF Be(CH)$_2$-I

(b) Infrared Spectra: $Be(OH)_2$-I

Infrared spectra obtained with pellet $Be(OH)_2$-I from room temperature to 500° after various times of heating are shown in Figure 2, facing. The spectra were measured with the pellet at room temperature, in sequence, after the various treatments tabulated below. The spectra 1-7 are displaced to avoid superposition.

	Evacuation Conditions	
Spectrum No.	Temperature, °C	Time, hours
1	room	1
2	400	1 1/2
3	400	3
4	400	8 1/2
5	400	26
6	500	1 1/4
7	500	2 1/2

The elimination of some bands, but only small changes in others, suggested that the decomposition was incomplete. These data will be considered in the summary of infrared data to be found on page 26.



IR. SPECTRA OF $Be(OH)_2$-VI

(c) Infrared Spectra: $Be(OH)_2$-VI

Infrared spectra obtained with pellet $Be(OH)_2$-VI at temperatures ranging from 400° to 850°C after various times of heating are shown in Figure 3, facing, the spectra were measured with the pellet at room temperature, in sequence, after the various treatments tabulated below.

	Evacuation Conditions	
Spectrum No.	Temperature, °C	Time, hours
1	400	1 1/4
2	700	1
3	700	3
4	700	5
5	850	1/6
6	850	2/3

After 40 minutes of heating at 850°C, the furnace melted. In order to allay suspicions that the decomposition was still incomplete, further experiments were made at 850° with pellet $Be(OH)_2$-XI. No further changes were detected. These data will be considered in the summary of infrared data to be found on page 26.

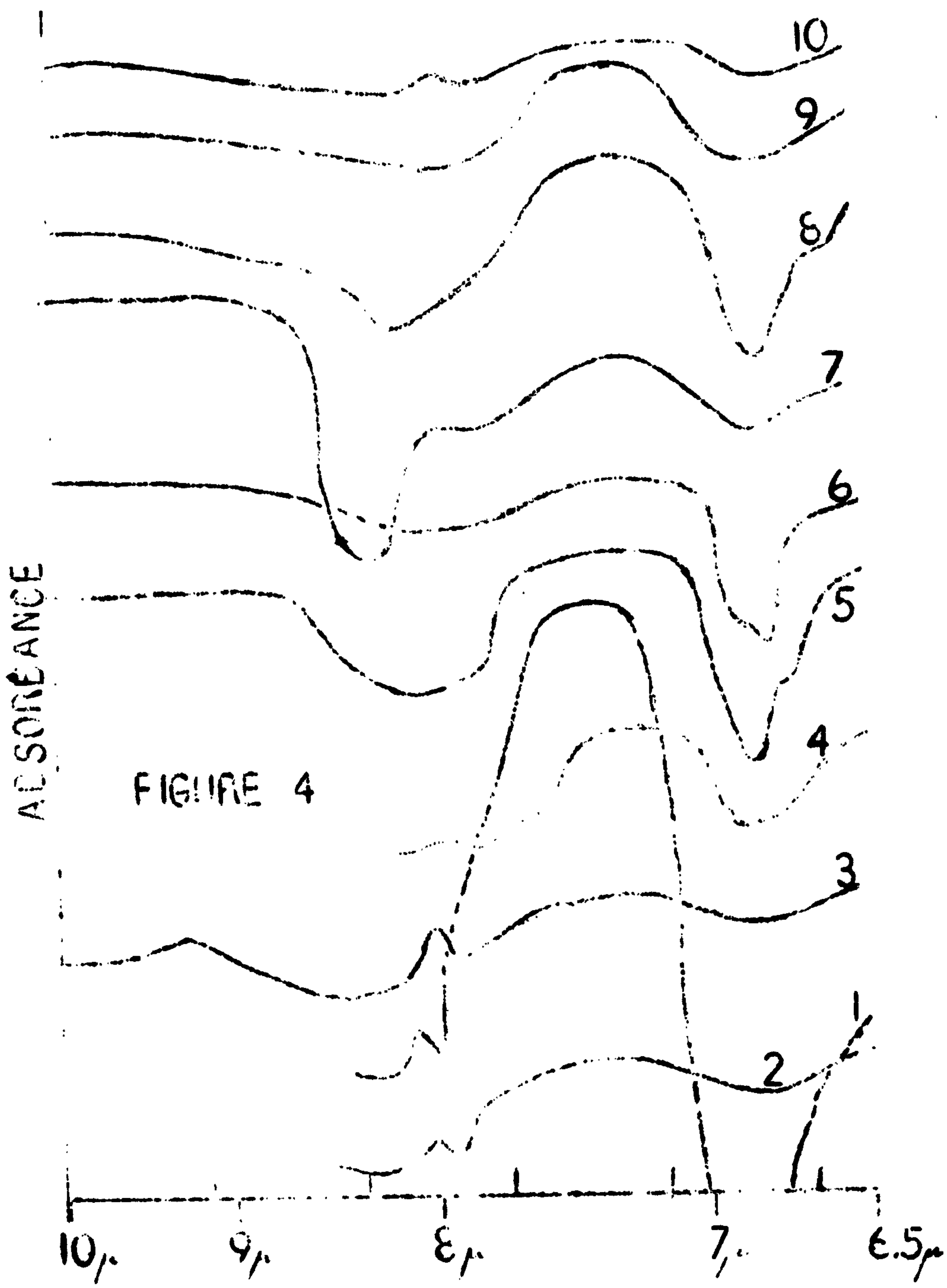


FIGURE 4

IR SPECTRA OF Be(OH)$_2$-XI

(d) Infrared Spectra: $Be(OH)_2$-XI

Infrared spectra obtained with pellet $Be(OH)_2$-XI at various temperatures and times of heating, and also after treatment with hydrogen, are given in Figure 4, facing. The spectra were measured with the pellet at room temperature, in sequence.

Spectrum No.	Treatment of Sample
1	Evacuation at 850° for 10 minutes
2	Evacuation at 850° for 1 3/4 hours
3	Evacuation at 850° for 5 3/4 hours
4	H_2 at room temp., at 1 cm H , for 10 min.
5	H_2 at 340°, 1 cmHg, for 2 hours
6	H_2 at 340°, 1 cm Hg, for 9 hours, cooled to room temp. and left in H_2 over night
7	Evacuated for 1 hour at room temp.
8	Evacuated at 400° for 5 hours
9	Evacuated at 700° for 1 hour
10	Evacuated at 850° for 15 minutes

(e) Color Changes of $Be(OH)_2$

Changes in color were noted to occur after the various heating and evacuation experiments. $Be(OH)_2$ and also BeO normally have a "white" appearance, i.e. they are colorless. It was noticed that $Be(OH)_2$ samples remained white, while being heated and evacuated, up to 400°. Above that temperature, however, a change occurred. In the region 500 - 700°C, the samples turned gray, while at 840 - 850° the samples became black throughout. A similar graying was noted with powdered BeO (II) samples after testing. The cause of these changes is not known, but might be related to a change in stoichiometry of the BeO.

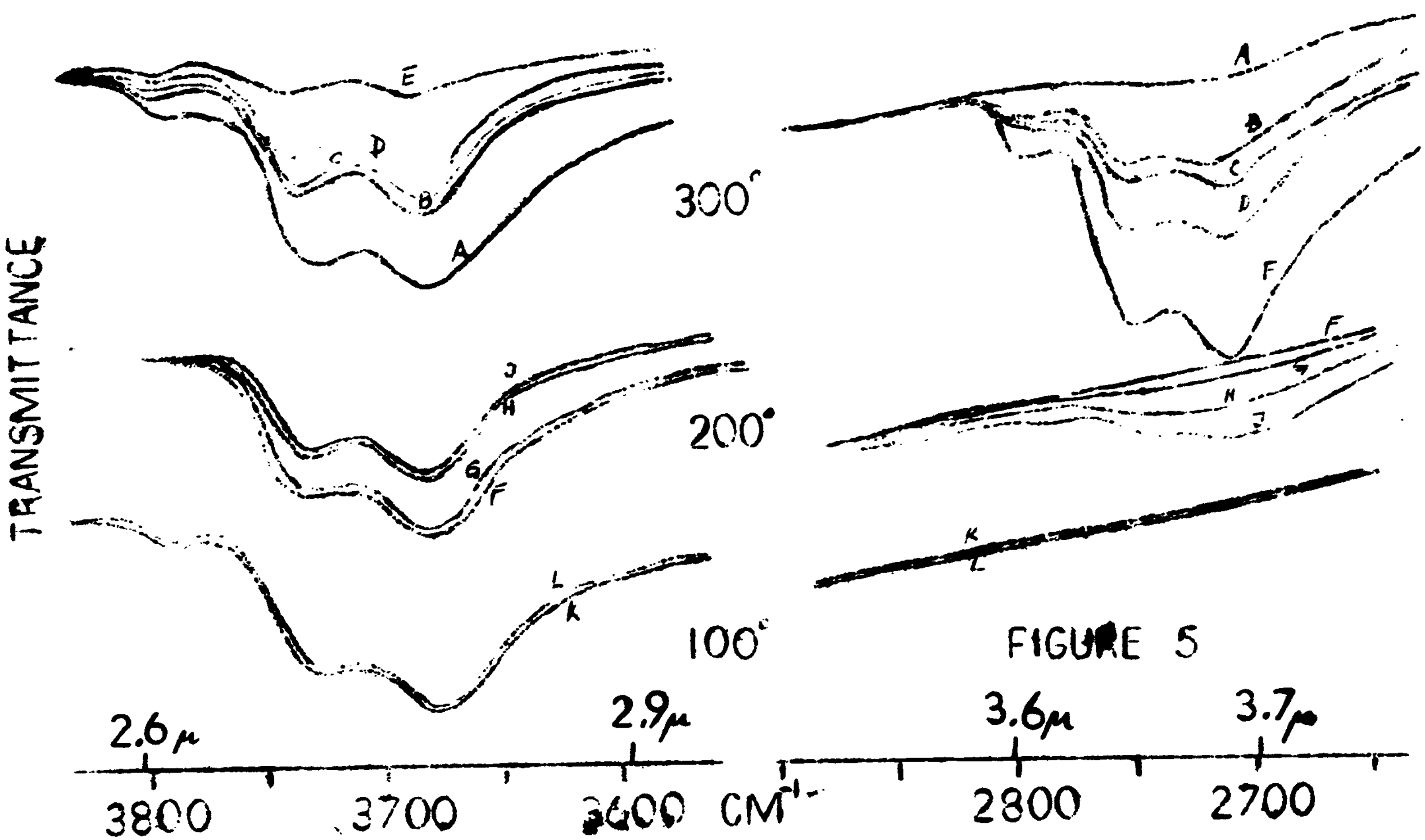


FIGURE 5

EXCHANGE OF OH GROUPS
ON Al_2O_3-III

6. Hydrogen - Deuterium Exchange on Alumina Surfaces

During the operation of a high-power electron tube, some of the metal of the electrodes is deposited on the tube walls. It is possible that this deposited metal enhances the uptake of hydrogen by the tube wall material. This hypothesis is being tested by investigating the replacement of the hydrogen of surface OH groups on alumina by deuterium.

(a) Samples

Samples were prepared from Alon-C gamma alumina. The powdered material was slurried with water to form a paste. The latter was dried and pressed into a 1 inch diameter disk containing 22 mg/cm^2_2 at 5 tons per square inch. This is designated Al_2O_3-III.

A second sample, designated Al_2O_3-1% Pt, was made by slurrying the alumina powder with a solution of chloroplatinic acid to yield a sample containing 1 weight per cent platinum. The metal platinum was chosen rather than the nickel typical of electrodes, because nickel is difficult to reduce in the presence of alumina.

(b) Infrared Spectra

Figure 5, facing, shows the results of exchange experiments. Al_2O_3-III was degassed in vacuum at 600°, and spectrum A was obtained. Deuterium was then introduced at 300° and after various times, spectra B,C,D and E were obtained. Deuterium was then replaced with hydrogen at 600 mm Hg pressure, and the cell was then pumped out, and spectra F was obtained. Spectra G, H, and J were then obtained after introduction of deuterium. Hydrogen was then introduced and, after degassing, spectra K was obtained. Spectra L was obtained

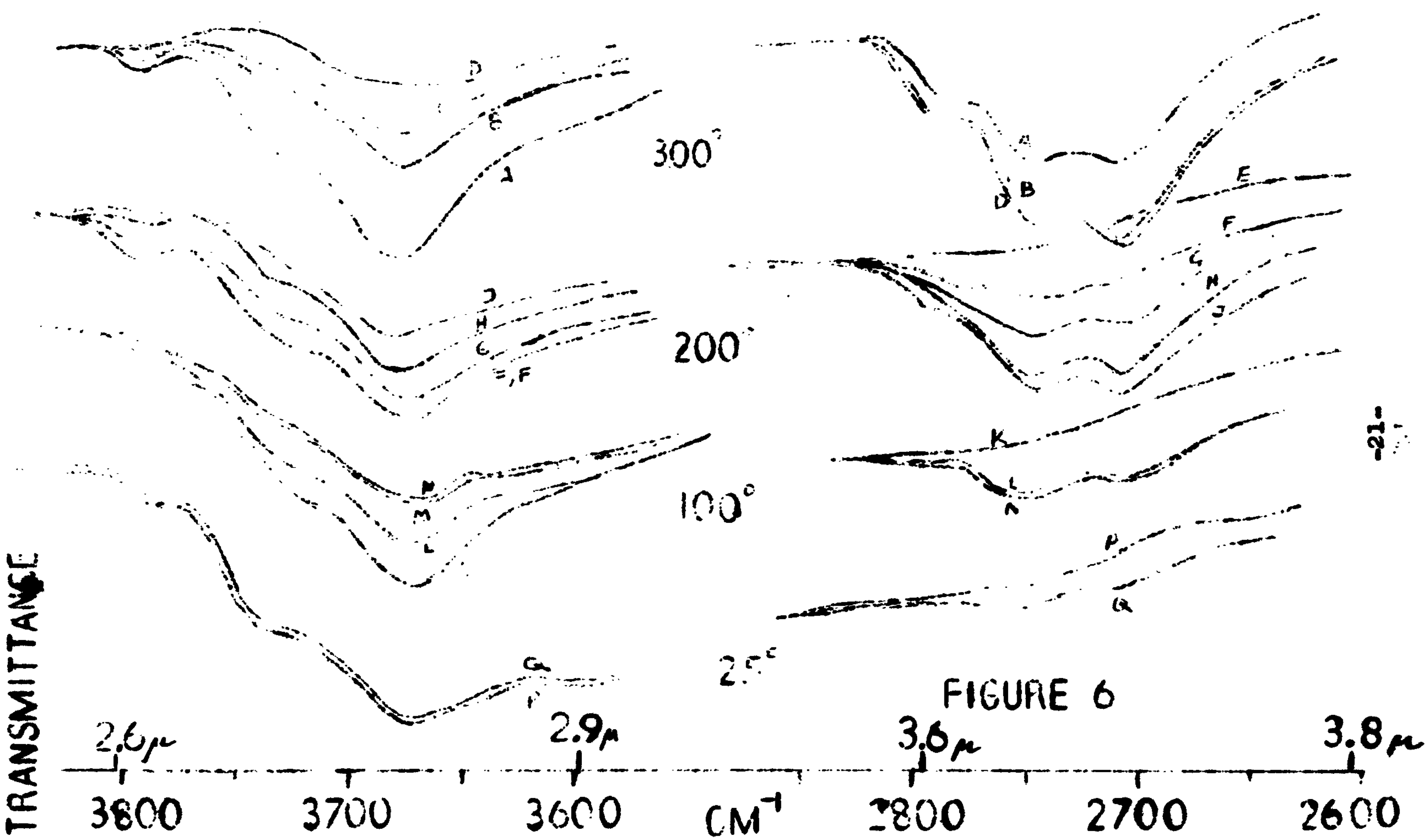


FIGURE 6

EXCHANGE OF OH GROUPS ON AL_2O_3-1% Pt

after introduction of deuterium.

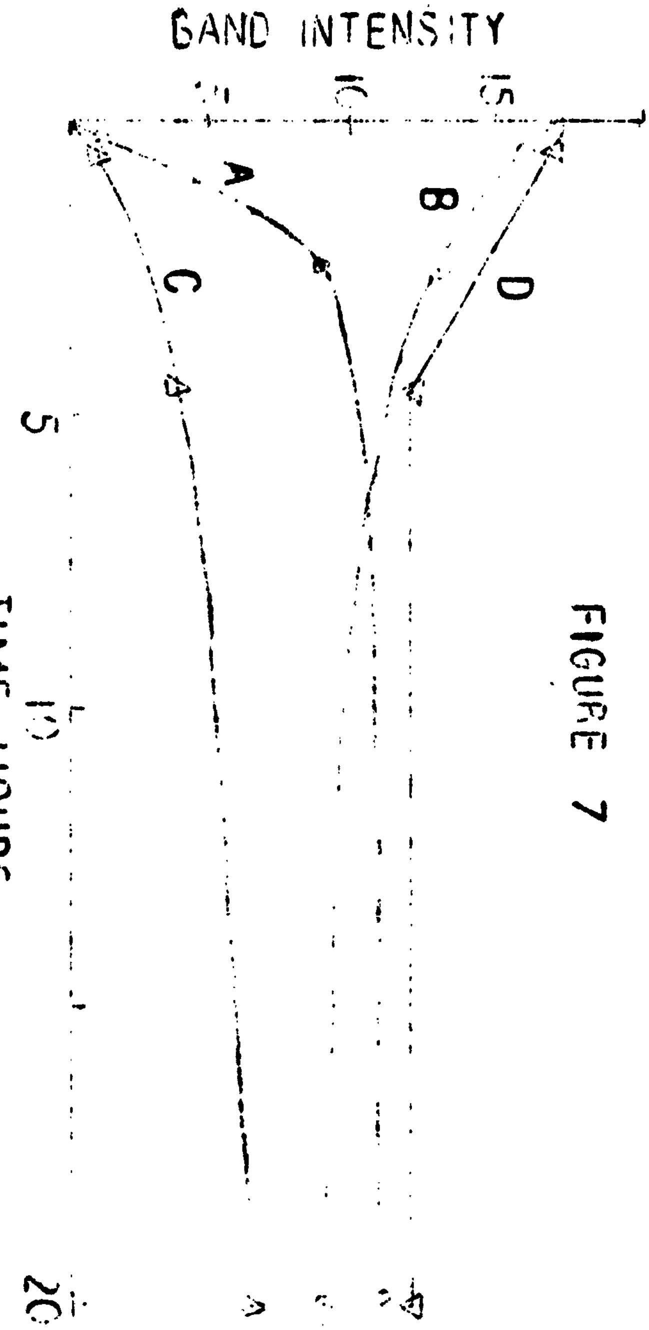
Each spectrum consists of two parts, one covering the region 3600-3900 cm^{-1} corresponding to OH group stretching vibrations; the second covering the region 2600-2900 cm^{-1} corresponding to OD group stretching vibrations. The series of the experiments corresponding to the spectra are tabulated below.

Spectrum	Exchange Time Time of exchange, hours	D_2 Pressure mm HG	Temp., °C
A	0		
B	1/2		
C	1	493	300
D	2 1/2		
E	47		
F	0		
G	1/2	485	200
H	4 1/2		
I	23		
K	0		
L	3 1/2	485	100

A second series of experiments were made with Al_2O_3-1% Pt. The experimental conditions and sequence of operations were the same as those for the experiments with Al_2O_3-III. The spectra are shown in Figure 6, facing. The experimental conditions are tabulated below.

Spectrum	Exchange time, hours	D_2 Pressure mm Hg	Temp., °C
A	1/6		
B	1/2	481	300
C	2 1/2		
D	24		
E	0		
F	1/6	491	200
G	1/2		
H	2 1/2		
I	19		

FIGURE 7



RATE OF OH GROUP EXCHANGE

K	0		
L	1/2		
M	2 1/2	499	100
N	17 1/2		
P	0	494	25
Q	1		

(c) Summary

Both samples of alumina, Al_2O_3-III and Al_2O_3-1% Pt, showed three bands attributed to O - H stretching vibrations. On treatment with deuterium, some exchange occurred, and the corresponding O - D surface structures were formed. The band positions, and the isotopic shift factors are:

O - H		O - D	factor
3795 cm^{-1}	shifted to	2795 cm^{-1}	0.735
3730 cm^{-1}		2750 cm^{-1}	0.737
3680 cm^{-1}		2705 cm^{-1}	0.735

The factor values are close to the theoretical value of $(2)^{-1/2}$.

Inspection of the data shows that no exchange occurred at room temperature with either sample. No exchange occurs with pure alumina at 100°, whereas exchange does occur at 100°, with the sample containing platinum. Exchange on Al_2O_3-1% Pt occurs at lower temperatures, and at a faster rate, than on Al_2O_3-III.

These relations, for example, are shown graphically by the plots of Figure 7, facing, of the intensities of the 2705 and 3680 cm^{-1} bands after various times at 200°C. The curve designations are as follows:

Curve	Sample	Band
A	Al_2O_3-1% Pt	2705 cm^{-1}
B		3680 cm^{-1}
C	Al_2O_3-III	2705 cm^{-1}
D		2680 cm^{-1}

Inspection shows the growth of 2705 cm^{-1} band, corresponding to an OD group, to be faster with Al_2O_3-1% Pt than with Al_2O_3-III. There is a similar difference in the rate of decline of the 3680 cm^{-1} band corresponding to an OH group.

A tentative interpretation of the effect involves the function of the metal on the surface. The two alumina samples contain approximately equal numbers of OH groups dispersed on the surface. Schematically, this can be represented as follows:

```
H                                  H
|                                  |
O                                  O
|       ( Pt   Pt )                |
AL      AL   AL   AL    AL    AL   AL
```

The H atom bonded to the O atom is exchanged with a D atom, so than an OD group forms on the surface. Note that the infrared spectroscopy detects the H-O bond, i.e. detects a signal from an OH group bonded to the surface of the alumina, and not from hydrogen bonded to the platinum. The suggestion can be made that the presence of the small metal particles on the alumina surface facilitates the exchange on the alumina surface by acting as centers of dissociation for hydrogen molecules.

It is tentatively proposed that hydrogen (or deuterium) molecules dissociate on the platinum particles. The resulting atoms then are able to leave the platinum and migrate over the alumina surface, to cause exchange of surface OH (or OD) groups. Essentially, there is a process of surface diffusion.

A series of experiments are planned to check this hypothesis by investigating aluminas containing less metal than Al_2O_3-1% Pt.

(f) Infrared Spectra: Summary

Several trends of change are noticeable in the various spectra of Figures 2, 3, and 4 on the thermal decomposition of $Be(OH)_2$ *in vacuo*. These can be summarized as follows.

1. After degassing at 400°C, three main absorption peaks at 6.4, 6.8, and 7.3 microns are observed. A smaller peak occurs at 4.3 microns.
2. After heat treatments in the range 500 - 700°C, the peaks at 6.4 and 6.8 microns disappear.
3. On heating at 850° the peak at 7.3 microns disappears. Peaks at 3.5 and 8.0 microns appear.
4. On the treatment of the sample, after heating at 850°C, with hydrogen at room temperature, 340°, and 400°, the 8.0 micron peak is not observed, but the peak at 7.3 microns, which had been removed by heating, gradually reappears. The 7.3 micron peak remains even after degassing at 700° and disappears only on evacuation at 850°, when the peak at 8.0 microns appears.
5. No peaks were to be found in the vicinity of 2.7 to 3 microns, the region "typical" of hydroxyl group .

Inspection of the various spectra suggests that the thermal decomposition of $Be(OH)_2$ occurs in the region 400-700°, but heating to 850° is necessary for complete decomposition to occur. After the 850° heat treatment the spectra obtained were similar to those

for BeO (J. R. Durig, R. C. Lord, W. J. Gardner, and L. H. Johnston, J.Opt.Soc.Am. 52,1078 (1962)).

The spectra of Figure 4 suggest that an interaction between hydrogen and the surface occurs. The high temperature necessary to cause changes in spectra of surfaces that had been exposed to hydrogen suggests that the study of hydrogen sorption by BeO requires degassing at or above 850°. In view of this, a series of experiments to study quantitative hydrogen sorption by BeO ex hydroxide after 850° degassing is planned.

It is interesting to note that no evidence was found for the formation of surface hydroxyl groups of the type known to occur, for example, with SiO_2 or Al_2O_3. Although such negative evidence is unsatisfactory, the absence of surface OH suggest that a sorption mechanism involving dissociation of the hydrogen molecule to form one surface OH and one surface BeH group can be ruled out.

An interpretation of the various absorption peaks is not given at this time. This would be premature and of dubious value, because further work must be done. It is expected that the work on the surface properties of BeO can be completed during the next report period.

CONCLUSION

1. Infrared spectra of the processes occuring during the thermal decomposition of $Be(OH)_2$ were obtained.

2. Infrared spectra of processes involving the interaction of hydrogen with BeO were obtained.

3. Infrared spectra of the exchange of hydrogen of the surface OH groups of alumina with deuterium to form surface OD groups were obtained. The presence of platinum on the alumina surface enhances the exchange process.

4. Further work is necessary on the above topics.

PROGRAM FOR THE NEXT INTERVAL

The following program is planned for the next interval:

1) Resume experiments designed to measure quantitatively the uptake of hydrogen on alumina under discharge conditions.

2) Continue infrared experiments on the exchange of surface OH groups, using an alumina sample containing 0.01% of Pt.

3) Continue infrared experiments on the interaction of H_2 and of D_2 with BeO.

PERSONNEL LIST

Name	Approximate Hours Spent on Contract
M. J. D. Low	150
N. Ramasubramanian	700
M. Courtois	700
D. J. Shombert	50

DR. D. J. SHOMBERT

Dr. Shombert is Assistant Professor of Physical Chemistry at Douglass College, Rutgers, The State University, and has joined the project. He has knowledge of and experience with surface problems as well as with electronic and electrical measurements. His interest in the general problem area has prompted his joining the project as a consultant, on a no-cost basis.

AD ____________

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2. Thyratrons, Super-Power Switches

I. U.S.Army Electronics R and D Lab.

II. Contract No. DA36-039-AMC-02170(E)

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CONTRACT DA36-039 AMC-02170(E)
Rutgers, The State University

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